From: Cynthia Caporale/ESC/R3/USEPA/US
Sent: 4/5/2012 5:26:18 PM

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Subject: Re: Verification/Completeness Check for Dimock (R# WO 1202003 PART 2 Posted Mar 20 and WO 1202004 PART 2 Posted Mar 21)

The report on the Dimock Verification/Completeness Check for file 1202003 FINAL Part 2 of 3 R33907 03 19 12 1713.pdf and 1202004 FINAL PART 2 of 3 R33907 03 21 12 1435.pdf were reviewed and below are the responses for your consideration.

File 1202003 FINAL PART 2 of 3 R33907 03 19 12 1713.pdf

1. For VOCs, the following qualifications should be applied to the following samples as noted based on the blank results (method, field, trip in that order) in accordance with the National Functional Guidelines: Acetone 3.3U for HW15a-P and HW31-P, acetone 4.0U for FB11 and FB12, acetone 4.6U for FB13, acetone 2.0U for HW51-P and HW43-P and acetone 3.2U for EB02; chloroform 0.5U for HW15a-P, HW15a, EB02, FB11, FB12 and FB13; toluene 0.5U for FB11and FB13; toluene 0.6U for FB12; and o-xylene 1.0U for EB02, FB11, FB12 and FB13.

Response: **Elevating the QL and qualifying "U" is not the typical procedure for R3 validation; however, if appropriate for this project we support that decision.**

2. For VOCs, the MS/MSD recoveries for bromomethane (48%/54%) for sample HW45 were outside the 70-130% criterion. Bromomethane results for sample HW45 should be qualified "UJ" in the Scribe result column.

Response: **When all other quality control information is within criteria, non detects are usually not qualified by R3 Lab for low LCS or MS/MSD recoveries; however, if appropriate for this project we support that decision.**

3. For VOC analysis, there doesn't appear to be any accuracy data for cyclohexane, Freon 113, methylacetate, methylcyclohexane or MTBE printed on the laboratory report. It appears that these compounds were added to the MS. All compound recoveries were calculated and within the QC criterion. No additional qualifications are necessary.

Response: **Not all LCS compounds are reported in the lab report due to issues with the Laboratory Information Management System; however, this information is available in the case files. We do not have an LCS for Freon 113, methyl acetate, MTBE, cyclohexane, or methyl cyclohexane. These compounds are part of the matrix spikes. Recoveries for all 5 compounds are within limits of 80-120%. We agree no qualification is necessary.**

4. For glycols, there is a statement in the case narrative stating that the blank spike results for two parameters were outside of quality control acceptance limits. All blank spike results were within QC criteria. This statement may be a carryover from a previous report. No additional qualifications are necessary.

Response: **We agree with this comment. The case narrative statement is a carryover from a previous report and should be removed. A supplemental report can be provided upon request.**

5. For SVOCs, the following qualifications should be applied to the following samples as noted based on the blank results (method, field, in that order) in accordance with the National Functional Guidelines: bis(2-ethylhexyl)phthalate 5.00U, diethyl phthalate 5.00U and di-n-butylphthalate 5.00U for HW45, HW45-P, HW43-P, HW43, HW15a-P, HW31-P, HW30, HW30-P, HW31, FB11, HW31z, HW15a, HW38-P, FB13, FB12, HW47, HW51, HW38, HW51-P and HW47-P; and diethyl phthalate 5.00U and di-n-butyl phthalate 5.00U for EB02.

Response: **Elevating the QL and qualifying "U" is not the typical procedure for R3 validation; however, if appropriate for this project we support that decision.**

6. This reviewer agrees with the raising the reporting limits to 60 µg/L for 4-chloroaniline, 2-methoxyethanol, and 3-nitroaniline for Batch BB20801 based on the acceptable recoveries in the mid-level spike for samples HW45, HW45-P, HW43-P, HW43 and EB02. No additional qualifications are necessary.

Response: **We agree with this comment.**

7. In the case narrative, the first and second lines under SVOCs state "For samples 1202003-01 thru -05, quantitation limits are elevated for 2-methoxyethanol and 3,3'-dichlorobenzidine due to 0% recovery in the 5ppb LCS" and "Results for the mid-level quality control check are within acceptance limits; therefore Quantitation limits are raised to the mid-level value" contradict the third line "For samples 1202003-01 thru -05 data for 3,3' dichlorobenzene is rejected due to 0% recoveries in the low and mid-level spikes." Since 3, 3'-dichlorobenzidine is one of the compounds that is not included in the lab report, this reviewer can't verify which statement is correct. Qualification of the data is pending based in the response from the R3 lab.

Response: **The BS1 for 3,3'-dichlorobenzidine = 0%. BS2 3,3'-dichlorobenzidine = 3%. The "R" is correct and 3,3'-dichlorobenzidine should be removed from the first sentence.**

Below is the exact SVOA narrative for reference:
For samples 1202003-01 thru -05, quantitation limits and 2-methoxyethanol and 3,3'-dichlorobenzidine are elevated due to zero percent recovery in the low-spike quality control check (BS1). Results for the mid-level spike quality control check (BS2) are within acceptance limits; therefore, quantitation limits are raised to the mid-level value. For samples 1202003-01 thru -05 data for 3,3'-dichlorobenzidine is rejected and qualified "R"due to zero percent recovery in the low- and mid-spike quality control check. For samples 1202003-01 thru -05, quantitation limits for 3-nitroanline are qualified "UJ" due to low percent recovery in the low-spike quality control check. For samples 1202003-01 thru -05, quantitation limits for 4-chloroaniline are elevated due to very low percent recovery in the low-spike quality control check. In the report, only 16 compounds are reported for the blank spike quality control check samples. Quality control information about the additional compounds is available in the case file.

8. For SVOCs, this reviewer agrees with the lab qualification of "UJ" for 2-methoxyethanol and 1-methylnapthalene since these two compounds were not added to the spikes for Batch BB21003. The "UJ flag should be carried over into the Scribe result column for samples HW15a-P, HW31-P, HW30, HW30-P, HW31, FB11, HW31z, HW15a, HW38-P, FB13, FB12, HW47, HW51, HW38, HW51-P and HW47-P. It is also recommended that the RL for 2-methoxyethanol be raised to the mid-level spike of 40 µg/L.

Response: **We agree, however, the 2-methoxyethanol quantification limit should be raised to 60 ug/L based on the mid-level spike amount.**

9. For SVOCs, hexachlorobutadiene and nitrobenzene do not appear to have been added to the spikes for Batch BB21003. Data for all the samples in this batch (HW15a-P, HW31-P, HW30, HW30-P, HW31, FB11, HW31z, HW15a, HW38-P, FB13, FB12, HW47, HW51, HW38, HW51-P and HW47-P) should be qualified "UJ" in the Scribe result column for Hexachlorobutadiene and nitrobenzene.

Response: **Both compounds were added to the spikes. For BS1, nitrobenzene recovery is 84.4% and hexachlorobutadiene is 65.6% recovery spiked @ 40 ug/ml. For BS2, nitrobenzene recovery is 73.6% and hexachlorobutadiene is 48.4% recovery, spiked @ 5 ug/ml. All are within acceptance criteria.**

10. For SVOCs, one base neutral surrogate and one acid surrogate were outside QC criteria for Batch BB21003. Since the recovery of the acid surrogate (2-fluorophenol) is less than 10%, all acid compounds 4-chloro-3-methylphenol, 2-chlorophenol, 2,4-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 4,6-dintro-2-methylphenol, 2-methylphenol, 4-methylphenol, 2-nitrophenol, 4-nitrophenol, pentachlorophenol, phenol, 2,3,4,6-tetrachlorophenol, 2,4,5-trichlorophenol and 2,4,6-trichlorophenol) should be qualified unusable "R" in the Scribe result column. Since the NFG allows for one base neutral and one acid surrogates to be out providing their recoveries are ≥10%, no qualification of the base neutral data are required. A "U" flag should be carried over into the result column for all base neutral compounds (not listed above) with the exception of pyrene which has a "J" flag.

Response: **We agree with the application of the "R" qualifier due to the surrogate recovery values exceeding criteria (one base neutral and one acid surrogate being out does not automatically required a "R" qualifier based on R3 procedures; however, the other base neutral and acid surrogates, although within criteria, had very poor recoveries indicating poor extraction efficiencies). We agree with the "U" and "J" flags stated above.**

11. It is assumed that all required instrument QC (RSD, %D, minimum response factors, etc.) specified by the method was run and was either within the criteria listed in the EPA R3 SOPs or qualified based on any deficiencies.

Response: **This assumption is correct.**

The report on the Dimock Verification/Completeness Check for file 1202004 FINAL Part 2 of 3 R33907 03 28 21 1435.pdf was reviewed and below are the responses for your consideration.

File 1202004 FINAL PART 2 of 3 R33907 03 21 12 1435.pdf

1. For VOCs, the following qualifications should be applied to the following samples as noted based on the blank results (method, field, trip in that order) in accordance with the National Functional Guidelines: Acetone 2.0U for samples HW48, HW48z, HW23-P, HW23, HW22-P, HW36n, HW16, HW44 and HW49; acetone 4.5U, bromodichloromethane 0.5U, 2-butanone 2.0U, chloroform 0.5U, toluene 0.5U and o-xylene 1.0U for sample FB14; toluene for samples HW54-P and HW49; and bromodichloromethane 0.5U, 2-butanone 2.0U, chloroform 0.5U, toluene 0.5U and o-xylene 1.0U for sample FB15. The RL for acetone for sample FB15 should be raised to 4.6 due to the TB.

Response: **Elevating the QL and qualifying "U" is not the typical procedure for R3 validation; however, if appropriate for this project we support that decision.**

2. For VOCs, the bromomethane recovery for the MSD (67%) was outside of the QC criterion for HW44. Bromomethane results for sample HW44 should be qualified estimated "UJ".

Response: **When all other quality control information is within criteria, non-detects are usually not qualified by R3 Lab for low LCS or MS/MSD recoveries; however, if appropriate for this project we support that decision.**

3. For VOC analysis, there doesn't appear to be any precision and accuracy data for cyclohexane, Freon 113, methylacetate, methylcyclohexane or MTBE for the LCS or the matrix spikes listed in the laboratory report in Batch BB22108. It is recommended that results for these compounds for all samples in this data set be flagged as estimated "UJ" (HW48, HW48z, TB31, HW21, HW21z, TB33, HW23-P, TB32, HW22, HW23, HW22-P, TB34, HW36n, HW49, HW16-P, HW54-P, FB14, HW16z, HW16, HW44, HW49-P, HW36n-PFB15, HW54, TB35, TB39, TB37TB38 and TB36).

Response: **Not all LCS compounds are reported in the lab report due to issues with the Laboratory Information Management System; however, this information is available in the case files. We do not have an LCS for Freon 113, methyl acetate, MTBE, cyclohexane, or methyl cyclohexane. These compounds are part of the matrix spikes. Recoveries for all 5 compounds are within limits of 80-120%. We agree no qualification is necessary.**

4. For VOCs, there is no case narrative in the laboratory report.

Response: **The VOC narrative was mistakenly omitted from the report. A supplemental report is available upon request. The narrative should have been submitted as follows:**

Acrylonitrile was analyzed on-demand using CLP equivalent methodology. This analyte does not appear in the data tables or the QC summary and all data for this compound is summarized here. Acrylonitrile was not detected in any of the samples above a quantitation limit of 2 ug/L. A four point curve was analyzed (2, 5, 10 and 20 ug/L). The samples were preserved to a pH<2 with HCl. A low level second source blank spike analyzed at a concentration of 2 ug/L had a recovery of 140%. A mid level second source blank spike analyzed at a concentration of 5 ug/L had a recovery of 95%. A matrix spike and matrix spike duplicate pair was prepared for sample 1202004-28 at a concentration of 5 ppb acrylonitrile with recoveries of 109% and 109%, RPD=0. A second matrix spike and matrix spike duplicate pair was prepared using sample 1202004-30 at a concentration of 5 ppb acrylonitrile with recoveries of 110% and 101%, RPD=9.

2-Chloroethylvinyl ether is not included in the analysis. 2-chloroethylvinyl ether breaks down in acidified samples.

5. For SVOCs, the following qualifications should be applied to the following samples as noted based on the blank results (method, field, in that order) in accordance with the National Functional Guidelines: bis(2-ethylhexyl)phthalate 4.76U, diethyl phthalate 4.76U and di-n-butylphthalate 4.76U for samples HW16-P, HW36n-P and FB15; bis(2-ethylhexyl)phthalate 5.00U, diethyl phthalate 5.00U and di-n-butylphthalate 5.00U for samples HW49-P and HW54; diethylphthalate 4.76U and di-n-butyl phthalate 4.76U for samples HW48, HW48z, HW49 and HW16; diethyl phthalate 5.00U and di-n-butyl phthalate 5.00U for HW21, HW23-P and HW36n-P; di-n-butyl phthalate 5.00U for samples HW21z, HW22-P and FB14; di-n-butyl phthalate 4.76U for sample HW16z; and bis-2-ethylhexyl phthalate 4.76U and di-n-butyl phthalate 4.76U for samples HW22, HW23 and HW44; bis-2-ethylhexyl phthalate 5.00U and di-n-butyl phthalate 5.00U for sample HW54-P.

Response: **Elevating the QL and qualifying "U" is not the typical procedure for R3 validation; however, if appropriate for this project we support that decision.**

6. For SVOCs in Batch BB21201, 4-chloroaniline (21%) was outside the QC criterion of 30-150% for LCS1 and 2-methoxyethanol (0%) was not recovered for LCS1. No mid-level spike was analyzed. Based on additional information supplied in the case narrative for Batch 21201, 3,3'-dichlorobenzidine and 2,4-dinitrophenol were not recovered in the low level spike. 4-Dinitro-

2-methylphenol was also recovered outside of the QC limits. The case narrative also indicated that pentachlorophenol was outside of the QC limits. Based on the information supplied in the report, pentachlorophenol was recovered at 18% and was within the 17-109% recovery and should not be qualified. Based on the information supplied, this reviewer agrees that the following samples should be qualified unusable (R) for 3,3'-dichlorobenzidine, 2,4-dinitrophenol and 2-methoxyethanol: HW48, HW48z, HW21, HW21z, HW23-P, HW22, HW23, HW22-P, HW36n, HW49, HW16-P, HW54-P, FB14, HW16z, HW16 and HW44. This reviewer also agrees that the following samples should be qualified "UJ" for 4-chloroaniline and 4,6-dinitro-2-methylphenol: HW48, HW48z, HW21, HW21z, HW23-P, HW22, HW23, HW22-P, HW36n, HW49, HW16-P, HW54-P, FB14, HW16z, HW16 and HW44.

Response: **We agree with this comment.**

7. For SVOCs in Batch BB21601, 4-chloroaniline recoveries were <10% and 2-methoxyethanol recoveries were 0% for LCS1 and LCS2. Based on additional information supplied in the case narrative, 3,3'-dichlorobenzidine and 3-nitroaniline recoveries were 0% in the low and mid-level spikes. 2,4-dinitrophenol and atrazine recoveries were outside QC limits in the low level spike but were acceptable in the mid-level spike. The case narrative also stated that 4,6-dinitro-2-methyl phenol and pentachlorophenol recoveries were low in the low-level spike. Based on the information supplied in the report, pentachlorophenol was recovered in the low level spike at 35%, was within the 17-109% recovery range and should not be qualified for sample HW49-P; the RL should remain at 5.00U. This reviewer agrees with the laboratory that results for 4-chloroaniline, 3,3'-dichlorobenzidine, 2-methoxyethanol and 3-nitroaniline be qualified unusable (R) for sample HW49-P. This reviewer also agrees with the laboratory that the RL for atrazine be raised to 60 µg/L and 4,6-dinitro-2-methylphenol be qualified "UJ".

Response: **We agree with all points.**

8. For SVOCs in Batch BB21501, 2-methoxyphenol was recovered in LCS1 at 0% and pentachlorophenol was recovered at 7%. Based on additional information supplied in the case narrative, 2,2'-dichlorobenzidine, 4,6-dinitro-2-methylphenol and 2,4-dinitrophenol were also recovered due to zero or low percent recovery in LCS1. The mid-level spike (LCS2) recoveries were within acceptance criteria. This reviewer agrees with elevating the RL to the mid-level concentration for these compounds (57.1 µg/L for samples HW36n-P and FB15; and 60 µg/L for sample HW54).

Response: **We agree with this comment.**

9. For SVOCs, the internal standard areas for sample HW54 were less than 50% of the area from the continuing cal or if run on an ICAL, the mid-point standard. This reviewer agrees with the qualification of "UJ" for sample HW54 only if the recoveries are within the 20-50% range for N-nitrosodimethylamine, benzaldehyde, phenol, bis(2-chloroethyl)ether, 2-chlorophenol, 2-methylphenol, acetophenone, bis(2-chloroisopropyl)ether, 1-methylnaphthalene, hexachloroethane, N-nitroso-di-n-propylamine and 4-methylphenol.

Response: **The internal standard recovery was 49%. We believe "UJ" is appropriate.**

10. For SVOCs, two acid and two base neutral surrogates were outside QC limits for sample HW21z. All data for sample HW21z should be qualified unusable "R".

Response: **We agree with the "R" qualifier for HW21z results.**

11. It is assumed that all required instrument QC (RSD, %D, minimum response factors, etc.) specified by the method was run and was either within the criteria listed in the EPA R3 SOPs or qualified based on any deficiencies.

Response: **This assumption is correct.**

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Subject: Verification/Completeness Check for Dimock (R# WO 1202003 PART 2 Posted Mar 20 and WO 1202004 PART 2 Posted Mar 21)

.....is attached for your review and consideration.

Ex. 4 - CBI
Lockheed Martin
Scientific, Engineering, Response and Analytical Services (SERAS)
Ex. 4 - CBI

[attachment "SERAS-172-DSR-032712_36.docx" deleted by Cynthia Caporale/ESC/R3/USEPA/US]